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Procedia Engineering 57 (2013) 739 – 745
Procedia Engineering
www.elsevier.com/locate/procedia

11th International Conference on Modern Building Materials, Structures and Techniques, MBMST 2013

Frost Resistant Ceramics Produced From Local Raw Materials and Wastes

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Abstract

The aim of this work was to analyse the possibilities to produce frost resistant ceramic body from local raw materials and wastes. Additional task is to investigate the influence of two waste materials (sawdust and milled windows glass) utilised together in the formation mass and burning temperature on the properties of ceramic masonry products. After analysis of research results, it was found that, it is possible to produce high frost resistivity ceramic with forecasted frost resistance larger than 1300 cycles and with effective porosity, determined after three days, lower than 6%. Moreover, by using local raw materials and additives, the porous frost resistant ceramics was obtained. The total open porosity of this ceramics reaches 35%, and forecasted frost resistance by beginning of disintegration is higher than 100 cycles.

Keywords: building ceramics; waste, frost resistance; porosity; compressive strength.

1. Introduction

Nowadays many researchers representing the science field of construction materials seek to utilize local raw and waste materials in order to produce frost resistant ceramic products, suitable for aggressive environmental conditions. Normally frost resistant ceramics is characterized by low water absorption (up to 5%) and higher strength.

It is very important to investigate the influence of burning regime on the frost resistance of ceramics, because the best burning temperature could help us to prepare frost resistant ceramic products [1], suitable to be exploited in very aggressive seashore climatic conditions. After the burning of ceramics at a determined temperature, scientists [2] have identified that, in order to produce frost resistant ceramic products, they have to be burned at the temperature not lower than 1055 °C. After the burning of samples at 1085 °C temperature, they successfully withstood more than 550 cycles. After 24 hours, water absorption of this ceramics reached 3.6%, and water absorption, identified through vacuuming, was 5.9%.

After series of experiments scientists [3] determined that for the estimation of frost resistance not only the parameters of porosity and pores' arrangement are significant, but also material's mechanical properties' allowing the ceramics with higher strength to withstand internal stresses, occurring when soaked water changes its form. In the research [4] it was found that clay's mineral composition has a large influence on frost resistance of the ceramic tiles. Frost resistance of the products produced from the clay with low amount of $CaCO_3$ is higher. The frost resistance achieved is worst due to the layered fabric of the phyllosilicates in the composition, this cause's large exfoliation in the tiles after extrusion [4].

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Selection and peer-review under responsibility of the Vilnius Gediminas Technical University
doi:10.1016/j.proeng.2013.04.093

Publications [5-7] include the relation between the maximal ceramics burning temperature (temperature range analysed 800°C–1100 °C) and operational frost resistance. After the increase of the burning temperature from 800 °C to 1100 °C, the values of operational frost resistance increase from 11 to 250 freezing-thawing cycles.

Researchers [8-10] analysed the influence of the maximal burning temperature on such properties of the final ceramic product as density, overall shrinkage, water absorption and strength. In [8] it was identified that, after the burning temperature is increased from 750 °C to 950 °C, density values of the ceramic body increase up to 11%, overall shrinkage – up to 56% and strength – up to 75 %. At the same time water absorption values decrease up to 8%. Scientists [9] found that, when maximal burning temperature is increased from 800 °C to 900 °C, density values increase by 10%, overall shrinkage – 5% and strength – 80%, and values of water absorption decrease from 25.61% to 0.64%. Authors [10] investigated the change of physical parameters of the ceramic body in the higher temperature range (1170 °C–1300 °C). After the ceramic semimanufactures were burned at the maximal burning temperature of 1250 °C and 1300 °C, X-ray analysis was carried out. The results of this analysis show that large amount of mullite and α – aluminates, and, in addition, hercynite spikes are already seen at the temperature of 1300 °C. After the identification of samples' physical properties, it was determined that overall porosity varied from 33% (when burned at the maximal temperature of 1170 °C) to 13% (when burned at the maximal temperature of 1300 °C), values of the density varied from 1300 kg/m^3 (when burned at the maximal temperature of 1170 °C) to 2940 kg/m^3 (when burned at the maximal temperature of 1300 °C), values of water absorption varied from 39.5% (when burned at the maximal temperature of 1170 °C) to approximately 0% (when burned at the maximal temperature of 1300 °C). Authors of the publication [11] showed that the density and strength of the ceramics increase when maximal burning temperature is altered from 1040 °C to 1080 °C.

Moreover, utilization of various waste materials is a big problem, and a large amount of waste materials could be used in the production of construction ceramics, because ceramic masonry products can be burned at 1000 °C and higher temperatures. In addition, exist waste materials that could improve the properties of ceramic body considerably.

Authors [12-13] analyzed the way how burning waste materials of sawdust, hydrolytic lignin, expanded polystyrene, rubber from used tires and paper production influence the values of density, porosity and compression strength of ceramic body. Following the findings drawn by the authors [12-13], we may conclude that the optimal amount of the additives analyzed in a formation mass is 15%.

Many scientists have investigated the influence of formation mixture additives, such as glass waste, ash, granite dust etc., on the density, strength, water absorption and other properties of the ceramic body. Authors [14] found that agglomeration process of ceramics starts at the lowest temperature when scrap glass is utilized. This indicates that melting glass, due to the increased reaction surface area, reacts more intensively with the clay and the maximal glass phase reaction degree can determine the formation of heat resistant elements, can increase the agglomeration interval as well as to stabilize the structure being agglomerated [15]. According to the conclusions of these publications, ground scrap glass influences the agglomeration of the ceramic body when 10% of scrap glass is added to the formation mixture.

Brazilian scientists [16] have carried out investigations with the aim to utilize eggshell waste for the production of ceramics. 4 formation mixtures were prepared, where the amount of eggshell waste was gradually increased by 5 percent, from 0 to 15%. It was identified that eggshell can be utilized in ceramics production, and the optimal amount of eggshell is 5–10%, when ceramic semimanufactures are burned at 1150 °C temperature. Density of such ceramics reaches approximately 1700 kg/m^3, water absorption – approximately 22%, and bending strength reaches 16 MPa [16]. Scientists from Czech Republic have determined that poppy seeds in the ceramics form more closed pores, and density of such samples reaches approximately 2500 kg/m^3, when the mixture is burned at 1570 °C temperature [17].

In the research [18], the change of the properties (density, chemical composition, mineral composition, strength) of ceramics with glass additive was analysed when the burning at the temperatures ranging from 600 °C to 1200 °C was applied. During this investigation ceramics with the strength of 90 MPa and density up to 2560 kg/m^3 was obtained.

The aim of this research is to analyze and determine the possibility to produce frost resistant ceramic body from local raw materials and wastes. Additional task is to investigate the influence of two waste materials (sawdust and milled glass) utilized together in the formation mass and burning temperature on the properties of ceramic masonry products.

2. Materials and techniques

Main raw material used in the research was the local clay. Chemical composition of clay is provided in Table 1, and granulometric composition – in Table 2. The chemical and granulometric compositions of this clay were determined by employing standard methods (LST EN 725-5: 2007, LST EN 1071-4: 2002 and others). The clay was passed through a 0.63 mm sieve.

Considering the results of X-ray analysis of clay (Figure 1), clay consists of the following minerals: kaolinite *K* (0.716, 0.450, 0.356 nm) ($Al_4[(OH)_8|Si_4O_{10}]$ or $Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O$), chlorite *X* (1.420, 0.716, 0.356 nm) $(Mg,Fe)_{6\text{-}n}$

$(Al,Fe)_n(OH)_8Al_nSi_{4-n}O_{10}$ n= 0.6-2, silica *Q* (0.426, 0.335, 0.246, 0.228, 0.213, 0.182, 0.167 nm) (SiO_2), dolomite *D* (0.289, 0.240, 0.219 nm) ($CaMg[CO_3]_2$,) calcite *C* (0.385, 0.304, 0.209, 0.191, 0.187 nm) ($CaCO_3$), illite (0.998, 0.500 nm)(K, H_3O) (Al, Mg, Fe)$_2$ $(SiAl)_4O_{10}[(OH)_2, H_2O]$ and small amount of feldspar *F* (0.324 nm) ($Na_2O{\cdot}Al_2O_3{\cdot}6SiO_2$). During the burning of the clay from Dysna deposit the following materials are formed from the initial clay minerals: hematite *F* (Fe_2O_3), gehlenite *G* ($2CaO{\cdot}Al_2O_3{\cdot}SiO_2$), anorthite *A* (CaO Al_2O_3 $2SiO_2$), diopside *D* (CaO MgO $2SiO_2$), cristobalite *Kr* (SiO_2), mullite ($3Al_2O_3$ $2SiO_2$), glass phase.

Cristobalite differs from the silica by its internal structure and has the other crystal structure. It is created when clay is burned at higher temperature. Feldspars consist of two minerals of isomorphic series – plagioclases and alkali feldspars.

Table 1. Average chemical composition of the clay

Chemical composition, %							
SiO_2	Al_2O_3+TiO_2	Fe_2O_3	CaO	MgO	K_2O	Na_2O	L.O.I.
51.63	20.09	7.56	5.45	1.46	3.35	1.15	9.10

Table 2. Average granulometric composition of the clay

Amount of sandy fraction (particles larger than 0.05 mm), %	Amount of dusty fraction (particles with the size ranging from 0.05 to 0.005 mm), %	Amount of clayey fraction (particles with the size ranging from 0.005 to 0.001 mm), %	Amount of clayey fraction (particles smaller than 0.001 mm), %
1.76	16.34	21.29	60.61

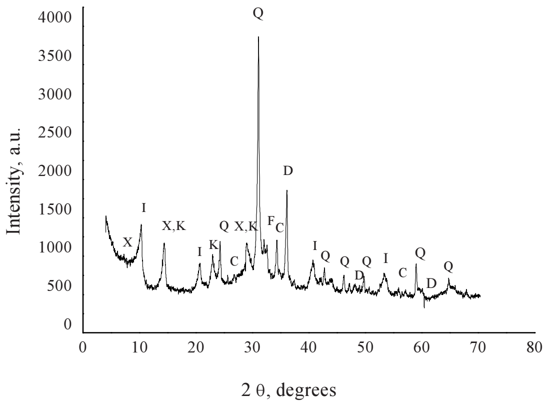

Fig. 1. X-ray pattern of the clay

In a first series, composition change occurs due to the exchange of heterovalent NaSi ↔ CaAl, in a second - due to the exchange of izovalent Na ↔ K. $K[AlSi_3O_8]$ and $Ca[Al_2Si_2O_8]$ formations do not intermix. Plagioclases form continuous row of solid solutions, from albite $Na[AlSi_3O_8]$ to anorthite $Ca[Al2Si_2O_8]$. They may consist of the impurity of $K[AlSi_3O_8]$ molecules, but only up to 10 %. Ca^{2+} easily replaces Na^+, because their ions' radii have similar length. Al^{3+} replacement by Si^{4+} compensates the charge difference. Stability zone of anorthite is very narrow. It is created at high temperature and is not resistant to the influence of fluids. Mica is sheet-shape mineral having volatile consistence and layered structure. Mica forms two main isomorphic rows: muscovite-pargasite – $K[Al_3Si_3O_{10}(OH)_2]$ – $Na[Al_3Si_3O_{10}(OH)_2]$ and biotite-phlogopite – $K_2[Fe5Al4Si5(OH)_4O_{20}]$ – $K_2[Mg_6Al_2Si_6(OH)_4O_{22}]$. In a first row isomorphism occurs due to the alternation of sodium and potassium, in a second – due to the alternation of iron and magnesium [19].

Nonplastics form a part of formation mass. These materials improve the drying process; decrease the shrinkage of the ceramic body during the drying and burning. As a result, stability of product's dimensions is increased. Better nonplastics are those which contain small amount of dust fraction (particles smaller than 0.1 mm), because this fractions lowers the strength of the semimanufactures [20]. Sand with average coarseness was used for the reduction of plastic clay. Chemical composition of this sand is provided in Table 3. In addition, the following additives were utilised: chamotte, sawdust and scrap glass of windows.

Table 3. Average chemical composition of sand

Chemical composition, %						
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	R_2O	L.O.I.
88.16	3.85	0.75	3.20	0.49	1.4	2.15

Table 4. Average chemical composition of glass

Chemical composition, %					
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	R_2O
71.18	1.64	0.21	8.21	3.26	15.5

Ground glass (its chemical composition is provided in Table 4) was introduced into the formation mass in order to help the melt formation at lower temperatures and to improve product's strength, frost resistance and other parameters. Scrap glass is inert waste material that is collected, separated, transported, stored as well as widely reprocessed and utilized in industry [21].

X-ray pattern of the chamotte utilized is shown in Figure 2.

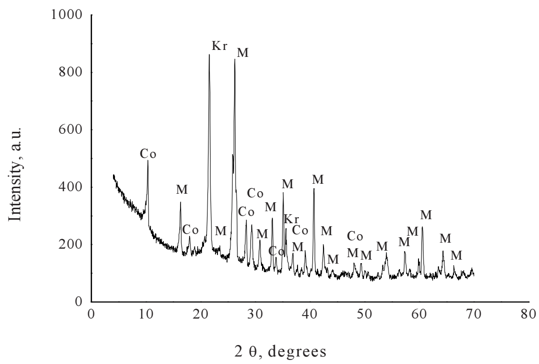

Fig. 2. X-ray pattern of chamotte (M – mullite, Kr – cristobalite, Co – cordierite)

X-ray pattern of the chamotte shows that the dominant mineral is mullite ($3Al_2O_3\,2SiO_2$) (0.542, 0.378, 0.339, 0.290, 0.270, 0.256, 0.243, 0.234, 0.230, 0.221, 0.213, 0.209, 0.189, 0.185, 0.180, 0.172, 0.170, 0.161, 0.158, 0.153, 0.146, 0.145, 0.141 nm), in addition, cristobalite (SiO_2) (0.411, 0.232 nm) and cordierite ($2MgO\,2Al_2O_3\,5SiO_2$), (0.850, 0.315, 0.304, 0.264, 0.243, 0.189, 0.180, 0.170 nm) exist. Mullite ($3Al_2O_3\,2SiO_2$) and cristobalite (SiO_2) are thermodynamically stable minerals, therefore, after the introduction of the chamotte (prepared from these minerals) into the mixture the properties of the ceramic products, especially exploitational frost resistance, should be improved. In addition, cordierite ($2MgO\,2Al_2O_3\,5SiO_2$), which is sufficiently inert to the temperature propagation, was found in chamotte. Products with large amount of cordierite are resistant to frost and high temperature.

The softwood sawdust was sieved through sieve 1.25 mm.

Table 5. Composition of formation mixtures and burning regimes

Batch	Amount of clay, %	Amount of sand, %	Amount of chamotte, %	Amount of sawdust, %	Amount of glass, %	Maximal burning temperature, °C
1	70.5	9.5	4.5	7	8.5	1090
2	66	9	4	13	8	1090
3	80	15	5	0	0	1090
4	70.5	9.5	4.5	7	8.5	1075
5	66	9	4	13	8	1075
6	80	15	5	0	0	1075
7	68	10	6	6	10	1075
8	66	9	4	13	8	1065

8 batches of samples (Table 5) were prepared to analyse and achieve research objective. These batches differed from each other by consistencies prepared and various burning regimes applied. Samples were prepared by applying manual and plastical method, by using the following raw materials: clay, quartz sand, chamotte, ground glass as well as wood sawdust (Table 5). After drying, the samples have been burned at different temperatures, which are given in Table 5: Temperature rise rate was 1.2 °C/min (maximal burning temperature 1090 °C) and 0.9 °C/min (maximal burning temperature 1065 °C and 1075 °C) maintaining at the maximum temperature for 5 h, the temperature decrease rate was 0.9 °C–1.2 °C/min. Temperature decreases to 400 °C in accordance with the set program. Later, the decrease of temperature is slower.

Prepared 70×70×70 mm samples were used to determine the physical-mechanical and structural parameters.

The samples prepared from the raw materials analyzed were dried until a constant mass was reached. The sintered and cooled samples were used to determine the physical-mechanical and structural parameters. The analysis of the powder was performed by using XRD. The main instrument used was difractometer DRON–7 with a cobalt anode, (wavelength $\lambda = 0.1792$ nm). The difractograms were decoded according to the characteristic values of distance between planes d and relative intensity I; values were presented in the standard tables [22]. The parameters of net dry density and water absorption of ceramic samples were determined in accordance with the standards respectively LST EN 772-13:2003 and LST EN 771-1+A1, compressive strength – LST EN 772-1. Exploitational frost resistance was forecasted according to the methodology of structural parameters [23-24], composed according to LST 1413.12:1998.

3. Test results and discussion

Values of the physical-mechanical and structural parameters are provided separately for each batch in Table 6.

The obtained values of physical-mechanical and structural parameters are rather different although mixture's consistence and burning regime varied slightly. Samples from batches 3, 6 were characterized by the highest density, reserve pore volume and overall shrinkage. This is an expected result, because burning out additive - sawdust was not added into these batches. The lowest density and reserve pore volume was obtained in the ceramic samples of batch 8. These samples included the largest amount of burning out additives – 13% and their burning temperature was the lowest – 1065 °C. After the burning of the semimanufactures with the same composition at higher – 1090 °C temperature, samples' density increased by approximately 16%, parameter of the reserve pore volume increased by 70%, relative wall thickness of the pores and capillaries increased – 60%, and capillary rate of mass flow decreased by almost 200%. In addition, compression strength of the samples from batch 2 increased considerably up to 16.36 MPa. Considering the results of the obtained parameters, it can be forecasted that the samples from batch 2 would be porous and sufficiently resistant to frost.

Table 6. Average values of physical-mechanical and structural parameters

Batch	ρ, kg/m^3	S_B, %	R_{gn}, MPa	R_p, %	D, %	g, g/cm^2	G_1, g/cm^2	G_2, g/cm^2	N, units
1	1552	13.07	14.26	55.78	1.86	0.75	1.11	1.13	0.10
2	1665	13.36	16.36	59.07	2.45	0.50	0.69	0.75	0.45
3	2106	14.79	23.89	64.47	8.59	0.13	0.38	0.39	0.70
4	1646	13.79	8.23	37.64	2,.54	0.91	1.19	1.19	0.18
5	1656	14.00	12.39	42.11	3.32	0.68	1.12	1.16	0.10
6	2077	14.79	14.82	54.96	8.01	0.30	0.56	0.56	0.32
7	1836	9.36	17.51	42.89	3.12	0.69	1.10	1.05	1.61
8	1430	13.14	8.02	34.74	1..53	1.44	1.80	2.05	0.15

ρ – density, S_B – general contraction, R_{gn} – compressive strength, R_p – reserve of porous volume, D – relative wall thickness of pores and capillaries, g – capillary rate of mass flow under normal conditions, G_1 – capillary rate of mass flow in a vacuum in the direction of freezing, G_2 – capillary rate of mass flow in a vacuum in the direction perpendicular to freezing, N – degree of structural inhomogeneity

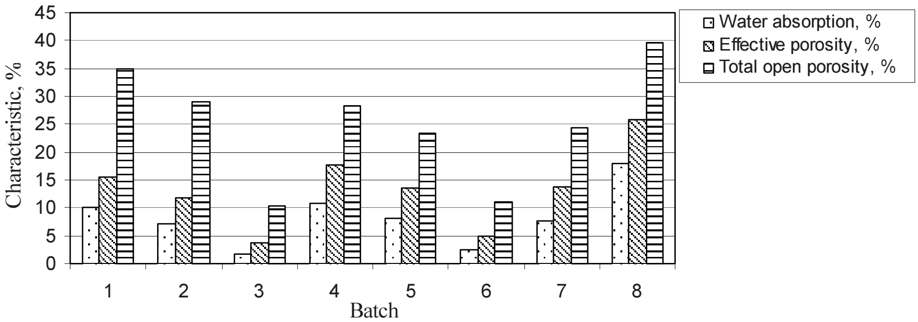

Fig. 3. Values of water absorption, effective and total open porosity by batches of samples

Figure 3 shows that low porosity ceramics was obtained and its water absorption after 72 h is lower than 3%. According to the reference [23] this ceramics belongs to the group I, because it's effective porosity is lower than 26%. Considering this data, the equations employed for the prediction of exploitational frost resistance are selected. This methodology of the prediction of frost resistance was developed by considering the long-lasting investigations according to standard LST 1413.12.

Results of the predicted exploitational frost resistance are provided in Figure 4.

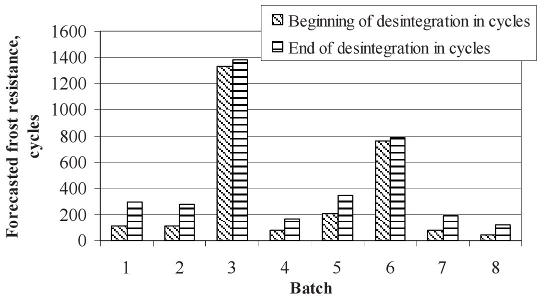

Fig 4. The beginning and end of disintegration in cycles by frost resistance of ceramic samples

Considering the results of investigation it was determined that the local clay is suitable to produce frost resistant porous ceramics. Such ceramics can be represented the best by the samples from batches 1 and 2, 5 because their porosity is approximately 30%, and frost resistance according to disintegration start is more that 100 cycles. According to the standards, this ceramics can be used at aggressive environmental conditions, and it has relatively low density. Heat conductivity ratio (estimated according to the density) of these batches is approximately 0.4 W/mK. Products of batches 3 and 6 can be distinguished by the highest frost resistance, because the clinker ceramics with water absorption smaller than 3% (after 72 h) was produced. Forecasted frost resistance (according to the beginning of fragmentation) of this ceramics reaches 1300 cycles. Reserve pore volume of the obtained clinker ceramics is rather high – 59.7%, this is the main parameter of exploitational frost resistance. This parameter indicates the amount of pores and capillaries where water penetrates very hardly. Average density determined for this ceramics is 2092 kg/m^3, and compressions strength is also rather high – 25 MPa. Clinker ceramics produced by utilizing typical raw materials has low (comparing the results of other researchers) burning temperature [25]. Scientists [25] produced clinker ceramics by burning the samples at the maximal burning temperature of 1210 °C. Our clinker ceramics was obtained by burning the samples at 1075–1090 °C temperature. In this way energy consumption is reduced considerably. Moreover, our clinker ceramics produced differs from others by the materials utilized for the production. In our case quartz sand and fireproof chamotte were utilized. In addition, all components were fined dispersive. Clay and quartz sand melted at high temperature and solid phase agglomeration took place together with liquid phase. Driving force of this agglomeration was melt's surface tension creating the negative pressure in a closed pore. Under the influence of this pressure melt filled the pores of ceramic material, and grains allied with each other. Larger amount of liquid phase and finer raw materials determine more intensive diffusion process in the sample. Due to this process grains of the material regroup, the amount of the open pores with irregular shape decreases, small, more closed, pores with more regular shape are created. Water hardly penetrates through the materials with this structure, during climatic changes water does not have a possibility to change its state, penetrate through and cause internal tensions, as well as fragmentation of the material.

4. Conclusions

It was determined that, when local clay is utilized as a main raw material, it is possible to produce high frost resistivity ceramics with forecasted frost resistance larger than 1000 cycles and with water absorption, determined after three days, lower than 3%. Such ceramics consists of the clay (80%), sand (15%) and chamotte (5%). Maximal burning temperature is 1090 °C.

By using local clay and other local raw materials, the porous frost resistant ceramics was obtained. The overall porosity of this ceramics reaches 35 %, and forecasted frost resistance is higher than 200 cycles. In one case, such ceramics was produced by utilizing 70.5% of the clay, 9.5% of quartz sand, 4.5% of chamotte, 7% of sawdust, 8.5% of ground glass. Maximal burning temperature was 1090 °C and time period for the storing at this temperature – 5 hours. In other case, the composition of samples' formation mixture was as follows: clay 66%, quartz sand 9%, chamotte 4%, sawdust 13%, and ground glass 8%. Maximal burning temperature is 1075 °C and time period for the storing at this temperature – 5 hours.

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